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Structure and NH_3 sensing properties of SnO thin film deposited by RF magnetron sputtering

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ABSTRACT

SnO thin films, 100 nm in thickness, were deposited on glass substrates by RF magnetron sputtering. A structure of SnO_2/SnO, where few nanometers of SnO_2 were determined on the SnO thin film by X-ray photoelectron spectroscopy. In addition, XPS depth profile analysis of the pristine and heat treated thin films was introduced. The electrical behavior of the as-sputtered films during heat treatment in air and nitrogen was recorded to investigate the working conditions for the SnO sensor. Subsequently, the NH_3 sensing properties of the SnO sensor at operating temperatures of 50–200 °C were examined, in which the p-type semiconducting sensing properties of the thin film were noted. The sensor shows good sensitivity and repeatability to NH_3 vapor. Finally, a sensing mechanism was proposed and discussed.

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1. Introduction

Stannous oxide is a p-type metal oxide semiconductor with a band gap ranging from 2.5 eV to 3 eV. SnO is a well-known coating material [1] and catalyst [2], and has recently been applied to thin film transistors [3–7]. However, gas sensing properties of stannous oxide is still lack of concern. Main reason for this problem may relate to the disproportion of SnO to Sn and SnO_2 ($SnO \rightarrow Sn_3O_4 + Sn \rightarrow SnO_2 + Sn$) at elevated temperatures [8]. This phenomenon has also been studied by Raman scattering, infrared (IR) reflectivity and X-ray diffraction (XRD) [9]. Moreover, a match between SnO (0 0 1) and SnO_2 (1 0 1) with similar Sn–Sn coordination (0.38 nm for SnO and 0.37 nm for SnO_2) may lead to conversion from the (0 0 1)-textured layers of SnO to the (1 0 1)-texture layers of SnO_2 [9–11]. Consequently, the layers of SnO_2 may be naturally formed on the surface of SnO. Thus, minimizing the thickness as well as the sensing property of SnO_2 layers on the surface of SnO is a key point to measure p-type semiconducting sensing properties of SnO.

The formation and expansion of the depletion and accumulation zones result in electron trapping and hole generation of the oxygen-adsorbed layers for n-type and p-type materials, respectively [12]. Therefore, the fluctuation of the material conductivity during heat treatment may provide us information about the formation priority of a depletion/accumulation zone at discrete points of treatment temperature. As the depletion zone plays an important role in the sensing properties of n-type semiconductors, selecting a right temperature where the formation/expansion of the depletion zone is weaker than those of the accumulation zone is a technique to measure the gas sensing properties of SnO.

In this study, SnO sensors were fabricated by depositing SnO thin films on glass-supported Ni/Au interdigitated electrodes using RF magnetron sputtering. The influence of temperature to the sensor resistance (R–T characteristic) was carried out to indicate the operating temperature of the sensors. Subsequently, NH_3 sensing properties of the sensor were examined using a dynamic gas testing system. Finally, a possible hypothesis for the behavior of the *R*–*T* curves during heat treatment and the sensing mechanism of the sensors are also introduced.

2. Experimental procedure

SnO thin films, 100 nm in thickness, were deposited by RF magnetron sputtering on Corning glass substrates and a Ni(40 nm)/Au(60 nm) interdigitated electrode (IDEs with electrode width: 100 μm and electrode spacing: 50 μm) at 200 °C. The sputter power, sputter rate and the ratio of Ar:O_2 were 50 W, 1.16 nm/s and 97:3, respectively. The total gas flow rate was modulated by mass flow controllers with the working pressure maintained at 10 mtorr. The *R*–*T* behavior and NH_3 sensing properties of the as-prepared samples were examined using a dynamic gas testing system. For the NH_3 sensing measurements, high-purity dry air was used as the base gas at a flow rate of 100 sccm. Before the measurements, the chamber was pumped to just below 8 mtorr to check for gas

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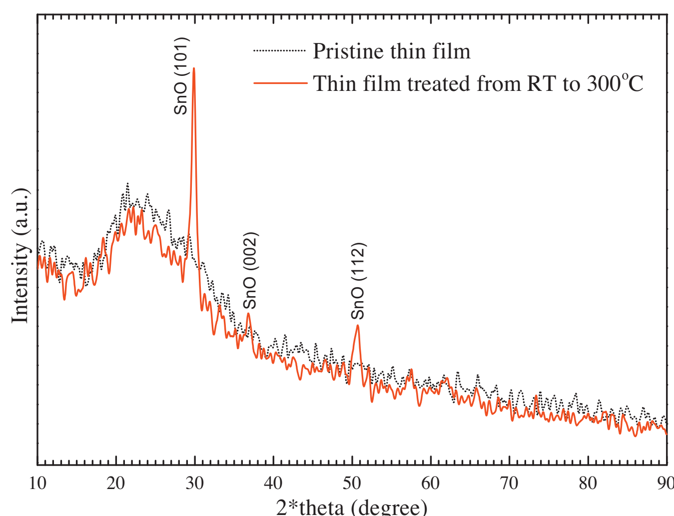

Fig. 1. XRD spectra of thin film before and after heat treatment (from RT to 300 °C).

leakage and remove contamination, such as moisture and residual gases. Dry air was used as the base gas during the measurements.

The surface morphologies of all samples were characterized by field emission scanning electron microscopy (FE-SEM: JSM-6701F). X-ray diffraction (XRD) measurements were performed on XPERT-PRO X-Ray Diffraction System using the CuKα_1 radiation (λ = 1.54178 Å) with a potential of 40 kV and a current of 30 mA. For the chemical composition, the depth profile and X-ray photoelectron spectroscopy (XPS) high resolution spectra were characterized by XPS analysis (Quantera SXM) in which the binding energy data were calibrated due to C_{1s} signal of ambient hydrocarbons (C—H and C—C) at 284.6 eV. Transmission electron microscopy (TEM) was performed on Telnai G2F20 S-TWIN, Philips.

3. Results and discussion

3.1. Structures of the thin film before/after heat treatment

XRD spectra of the pristine thin film and the film treated from RT to 300 °C are shown in Fig. 1. These spectra show the amorphous phase of the pristine thin film. For the film treated from RT to 300 °C, 3 peaks at 29.8°, 37.1° and 50.7° indexing to tetragonal structure of SnO (1 0 1), SnO (0 0 2) and SnO (1 1 2) (Reference: Joint Committee for Powder Diffraction Standard – JCPDS: 01-072-1012) were found. Besides, no impurity peaks were observed in those patterns.

Fig. 2 introduces FESEM images of the pristine and the treated film (from RT to 300 °C) which were deposited on IDEs. It can be easily seen in the figure that the thin film surfaces were constructed of grains. In addition, the grain sizes of both samples were similar (below 100 nm).

XPS depth-profiling of the as-deposited thin film was probed as a function of layer thickness (Fig. 3a). Following these data, about 19 sputtering cycles (nearly 19 min in total) were required to remove a 100 nm thickness of the pristine thin film and reach the glass substrate. Therefore, an approximate sputtering rate was calculated of 5.26 nm/min. The drop of C_{1s} concentration after 1 s sputtering indicated an elimination of the ambient hydrocarbons on the sample surface. The elemental distributions of Sn and O in the thin film can also be found in Fig. 3a where the Sn_{3d5} concentration increased from 50% to 62% and inversely, the O_{1s} concentration decreases from 50% down to 35%.

The high-resolution O_{1s} (Fig. 3b) and Sn_{3d5} (Fig. 3c and d) XPS spectra introduce in more detail the chemical composition of the pristine thin film followed the distance from the thin film surface. The O_{1s} spectrum could be Gaussian fitted to 3 peaks at 530.1 eV, 531.5 eV and 532.4 eV (Fig. 3b). On the thin film surface, the high band energy component at 530.3 eV indicated the formation of the SnO_2 phase [13]. Besides, the O_{1s} peaks at 531.7 eV and 532.6 eV were assigned to the presence of a hydroxyl groups and water

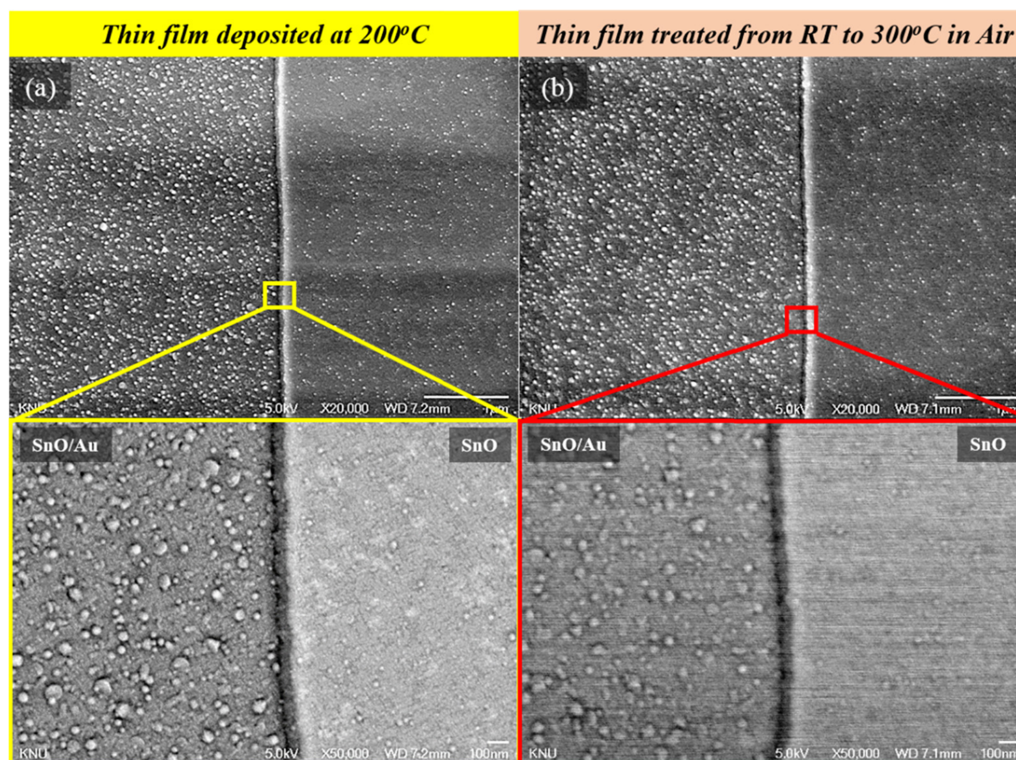

Fig. 2. FESEM images of the pristine thin film (a) and thin film treated from RT to 300 °C (b). (For interpretation of the references to color in the text, the reader is referred to the web version of the article.)

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